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FIELD TRIP TO LABORATORIES THAT DISPLAY SCANNING ELECTRON MICROSCOPES AND IMAGE ANALYSIS SYSTEMS

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March 1983

Project MRP-3.3.0.0.03
Mineralogical Studies of Marginal and Complex Ores

22 pp

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 83-44 (TR)

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by

W. Petruk and P.R. Mainwaring

SYNOPSIS

Five laboratories were visited in January and February, 1983 in order to evaluate image analysis systems that could perform the required image analysis in the MSL laboratory. The desired system would consist of a scanning electron microscope (SEM) to produce the image, an energy dispersive X-ray analysis (EDXA) system to identify the phases, and computer using image analysis programs to determine the quantities, sizes and shapes one- and two-phase grains in the image. The computer would steer the X-ray beam to individual grains to identify the phases and would perform the image analysis. The system would be fully automated to perform the analysis unattended. All the systems observed could meet the minimum requirements, but none have all the needed image analysis programs. Consequently the manufacturers have been asked whether they could make a commmitment to write a suitable program if we should choose their system. The current judgement is that the JEOL SEM equipped with a TRACOR NORTHERN EDXA system and image analysis program has the best potential of meeting our requirements. The evaluation, however, is continuing.

INTRODUCTION

Four laboratories were visited during the period of January 25-28, 1983, to evaluate SEM-EDXA-based image analysis systems. Thse systems consist of a scanning electron microscope (SEM), energy dispersive X-ray analyser (EDXA) and an image analyser. The laboratories visited wre Lemont Scientific Inc., 2011 Pine Hall Drive, Science Park, State College, Pennsylvania 16801, Phone (814)238-8403; U.S. Steel Corporation, Research Laboratory, Monroeville, Pennsylvania 15146; JEOL, U.S.A. Inc., 11 Dearborn Road, Peabody, Ma 01960, Phone (617)535-5900; and Bausch and Lomb, 820 Linden Avenue, P.O. Box 9189, Rochester, N.Y. 14625, Phone (716)338-8362.

LEMONT

The persons met at the laboratory were:
Dr. Jozef Lebiedzik - Vice-President,
Mr. Robert M. Edwards - Director of Software Development,
Mr. Paul Anderson - Marketing Manager.

The image analysis system is set up both as a demonstrator and a commercial unit. It consists of an ISI SEM, an EDXA and a Lemont image analyser (Figure 1).

To perform the analysis a chemistry file has to be set up by the operator in order to identify the phases. The file is based on X-ray intensities produced by each particle. For the demonstration they used a chemistry file that had been previously set up. This suggests that it takes a long time to establish a new chemistry file.

To prepare for analysis a backscattered electron image (BSE) of the sample is displayed on the CRT and a line is scanned across the image to produce a spectrum of the grey levels in the image. Thresholds are set to detect all grains that are either whiter or blacker than a certain grey level. All grains so detected are analysed. The instrument has both a diameter and a grindometer algorithm, but only the diameter analysis was demonstrated.

- To perform the analysis the field is scanned until a grain is detected; exploratory diameters are then set out to find the centroid of the detected grain. Enough diameters are drawn to define the particle,

and the area of each triangle so produced is calculated. The sum of the triangular areas is the area of the particle. The ends of the diameters are joined and the sum of these segments is the perimeter. During analysis, five particles are first defined by image analysis; the beam is then steered back to the centroid of each particle for analysis by EDXA to identify the chemistry of the particle. Hence the image analysis is performed first, particle identification next. The area shown in Figure 2 was analysed, at magnifications of about 150X and 270X. The magnification at 150X was too low and the centroids of many particles were not found when the beam was steered back for phase identification. At a magnification of 270X, all particles were detected and identified. One hundred ninety_five particles were analysed in 20 minutes or 1200 seconds, i.e., about 6 seconds per particle. The wrong chemistry class was set up, therefore, the data output was meaningless. Furthermore, the presentation of the data was not clear.

Comment: The system has the potential to perform the analysis required for our need, but not as the programs now stand. The deficiencies are:

(1) The time for analysis per particle is too long. A maximum of two seconds is required. Therefore a change in the basic program is needed.

(2) The format of the data output needs to be completely revised.

(3) The diameter analysis does not accurately define grains with complex shapes.

(4) The existing program cannot analyse and define grains that have two phases.

(5) The existing program does not have the facility to search for a particular mineral.

Mr. Edwards, Director of Software Development assured us that the required software can be written and that they are prepared to write it.

The cost of the Lemont image analysis system with required auxiliary features is $41,000. A suitable SEM and EDXA system is about $250,000, for a total of about $300,000.

U.S. STEEL

The personnel met at U.S. Steel were:

Dr. R.J. Lee,

Dr. J. Kelly,

Mr. N. Panseri.

There are several scanning electron microscopes in the laboratory. Two are equipped with Tracor Northern EDXA systems; one Tracor Northern EDXA system has a particle analysis program (image analysis) (Fig. 3). U.S. Steel personnel prefer the Tractor Northern particle analysis program to the Lemont system. They have a Lemont system in their laboratory but do not use it. The demonstration indicated that the Tracor Northern Image Analysis System operates like the Lemont system and has the same inherent problems with respect to our needs.

The other Tracor Northern EDXA system has a digitized colour coded X-ray mapping display, where the X-ray map of each element (up to 8 elements) can be displayed and a combination map of all elements can be produced, (Fig. 4).

JEOL USA INC.

The persons met at the JEOL Laboratory were:

Jean-Pierre Slakmon - Canadian representative,

Robert T. Santorelli - Manager, Sales Department,

J.D. Geller - Manager, Surface Analysis Laboratory,

Charles H. Nielsen - Manager, Applications Laboratory,

Jarry Shattack - Tracor Northern.

The JEOL Laboratory at Peabody is well equipped for demonstrations, analysis, and applications. They also make special arrangements for users to work for short periods at their laboratories on specific problems. The laboratory has several SEM's, a microprobe, an auger probe, and other equipment. The company uses the Tracor Northern EDXA system on their SEM and microprobe; thus the image analysis and beam steering are in the Tracor Northern system (Fig. 5 and 6). As stated above the image analysis program of Tracor Northern is written with an algorithm similar to the Lemont system; it suffers from similar deficiencies. However, the units at the JEOL laboratory appear to have a sub-routine that properly handles complex grains, and a sub-routine that can be used as a search. Furthermore, the image analysis routine

is much faster than the one of Lemont, because they draw fewer diameters. The only real deficiency is in handling two-phase grains. A request has been made for a commitment from their programmer to write a sub-routine for two-phase grains. An additional feature of the Tracor Northern image analysis program is that it is written in "flextran", a very easy computer language for the operator to use. The program has many features which we were unable to study or evaluate, due to lack of time.

The system JEOL 733 equipped with a Tracor Northern EDXA unit using an image analysis program has nearly all the features required for automatic high quality image analysis. The SEM has high resolution, excellent detection and phase discrimination, and an automated stage. Photographs showing the topography, BSE image, and X-ray images of our sample show some results (Fig. 7-12). The unit is the most expensive of all observed. The total package includng a program for analysing two-phase grains would be about $470,000. However, the capabilities and speed of analysis surpass the others.

BAUSCH AND LOMB

The persons met at the Bausch and Lomb laboratory were:
Don A. Nelson - Regional Sales Manager,
Dr. Kenneth J. Karcich - Applications specialist,
_____ - Chief programmer.

The demonstrator image analysis system in the laboratory consisted of a NANOLAB 7 SEM, an EDXA unit, and an Omnicon (Fig. 13). The Omnicon operates by scanning the grains and analysing each grain as it is scanned. Therefore, it has the potential of being quick. During demonstration, the program was set to stay 5 seconds on each grain in order to identify it, thus it took over an hour to analyse a field of 600 grains (about 6 seconds per grain). As stated above, this is too slow. The chemistry file that was used during the demonstration did not relate to the sample used, therefore, the results were meaningless. The printout of the results, was like the printout from other units, clumsy and difficult to comprehend.

EVALUATION OF ENERGY-DISPERSIVE DETECTORS AND COMPUTER SYSTEMS

There are two main requirements for an energy-dispersive system to be used most effectively as part of an automatic image analyser:

1) The system should be fully integrated into the image analysis system such that commands and data transfer are fast and to not degrade the analysis time and data transfer is instantaneous; the software should also be integrated with the image analysis software such that analysis of data and image analysis can be simultaneous.
2) The energy dispersive system should not duplicate any hardware.

Point 2) above is important because most automatic image analysis systems dedicate an entire computer to image analysis, usually a computer of different manufacture and utilizing a different computer language. Other systems use one computer which controls both the image and X-ray analysis.

During the field trip, systems from the four most prominent producers of EDXA systems were seen in operation (Kevex, Ortec, Princeton and Tracor). Some drawbacks in the image analysis systems are due to inherent limitations in the associated energy-dispersive analysis systems. Only Tracor Northern Inc. has produced a system which performs image analysis, X-ray analysis and data processing centered around a single, fully integrated computer (LSI-11/23). On other counts, the EDXA systems are quite similar in performance and specifications with respect to sensitivity and electronic stability.

Two systems have been examined which are capable of sophisticated image processing. These are Ortec "image-master" and the Tracor TN-2000 IPP system. This software would provide significantly enhanced capabilities for automatic image analysis systems. Unfortunately, at the present time the Ortec system does not have integrated software for particle analysis as well as image analysis. The Tracor system has this capability and Ortec is likely to develop this in the future.

At the present time, it appears that only Tracor Northern has the hardware and software developed for integration into an automatic image analyser.

All energy-dispersive detector systems which were demonstrated during this field trip were microcomputer-based. Furthermore, all utilized the 16 bits per word LSI II central processor which has become the industry standard for scientific computing and instrument control. This deep market penetration by DEC computing equipment implies that their software has also achieved recognition as the industry standard. In fact, the only EDXA system that does not rely on DEC hardware and software is the LINK system. For maintenance, upgrading and future expansion and software compatibility between laboratories it is strongly suggested that any future equipment purchases requiring a dedicated computer should be based on PDP-11 or LSI-11.

CONCLUSIONS

All the SEM-EDXA image analysis systems observed have the capability of steering the beam to a grain and of identifying the grain. The potential for performing accurate image analysis exists on all the systems. None of the systems, however, have all the programs required for our analysis, consequently additional programs would have to be written. A list of our requirements has been prepared and sent to each company (see appendix) with the request to reply whether they can write a program to meet our needs. The programmers at both Lemont and Bausch and Lomb have indicated that they can write the appropriate programs, but we want this in writing. Tracor Northern have not committed themselves at this time.

JEOL 733 equipped with a Tracor Northern EDXA has the best potential of meeting our needs. The other systems might fulfill our needs, but would not be as versatile and would be slower to operate.

The Ortec salesman has pointed out that the Ortec EDXA has an image master that can enhance the image; this could improve the image analysis. They use the Lemont image analysis program. A unit at the Department of Medicine, U. of T. was demonstrated Feb. 25, 1983. Unfortunately the enhanced image is a parallel with the image analyses program and hence is not available for image analysis with the existing program.

The exploratory trip clearly showed that the technology for a fully automatic SEM-EDXA based image analysis system that we require is developed to a greater extent than the literature indicates and that only a few computer programs need to be written to meet our needs. This is much better than was expected.

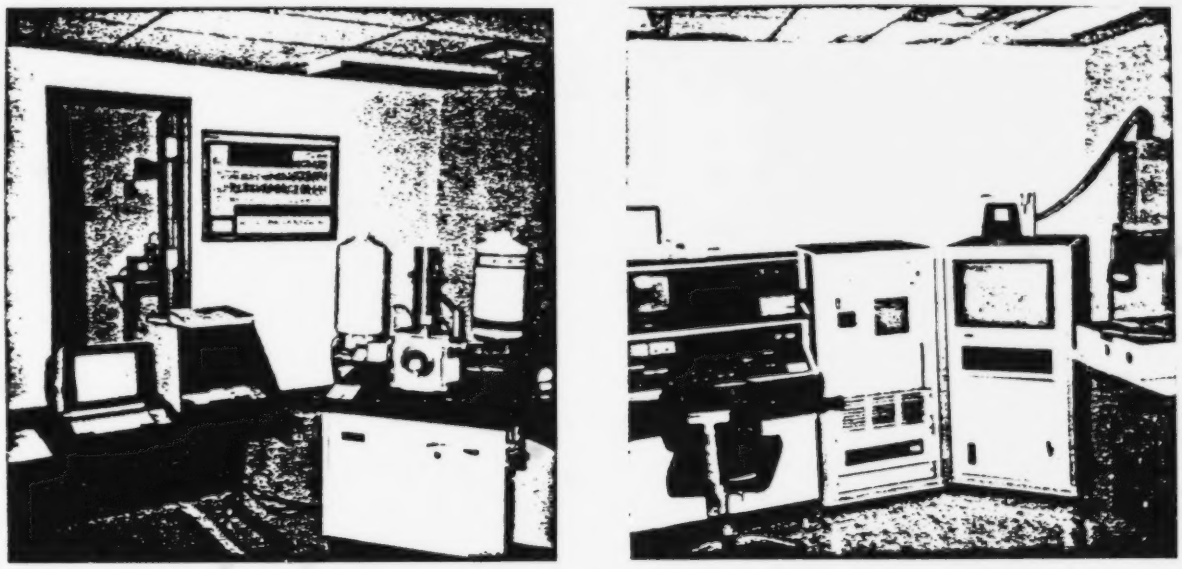


Fig. 1. Image analysis system at Lemont Laboratory. Right photo: Lemont image analyser on right, SEM panel on left; Left photo: ISI SEM gun and sample chamber, and Ortec EDXA; on left side CRT and keyboards for image analyser and EDXA.

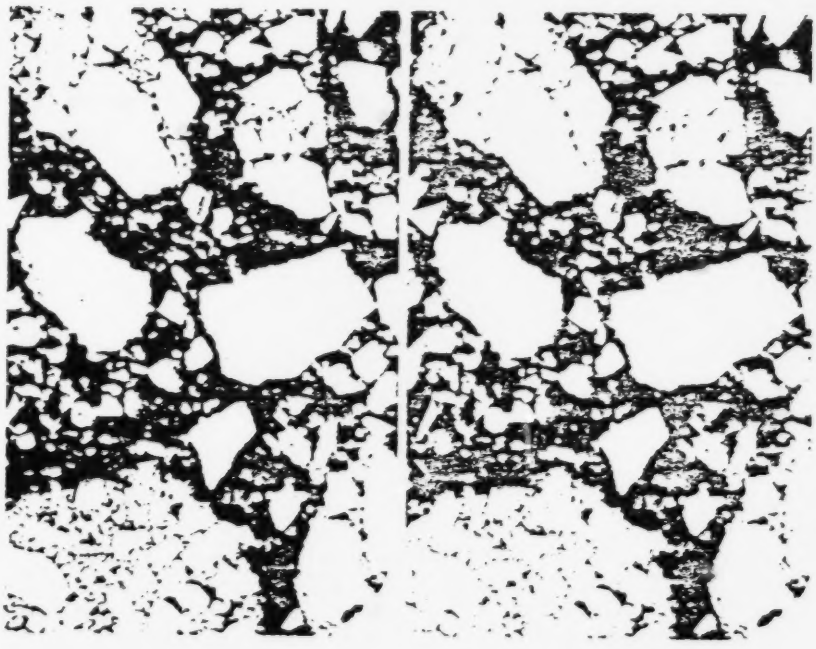
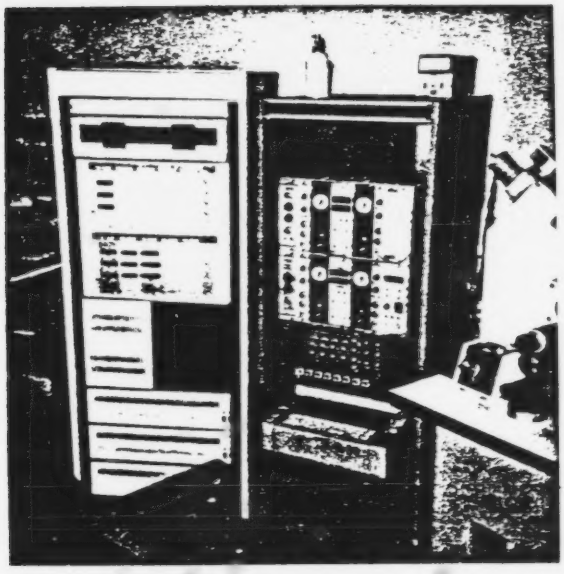


Fig. 2. Secondary image and BSE photomicrographs at 270X of Pb concentrate.

Fig. 3. Image analysis system at U.S. Steel. Right photograph, SEM panel and EDXA. Left photograph, CRT, keyboard and Tracor Northern cabinet and software.

Fig. 4. U.S. Steel; Right photograph, SEM and Tracor Northern EDXA with digitized X-ray mapping facility. Left photograph, X-ray maps for Fe, P, Ni and composite.

Fig. 5. Jeol SEM with Tracor Northern EDXA programmed to perform image analysis.



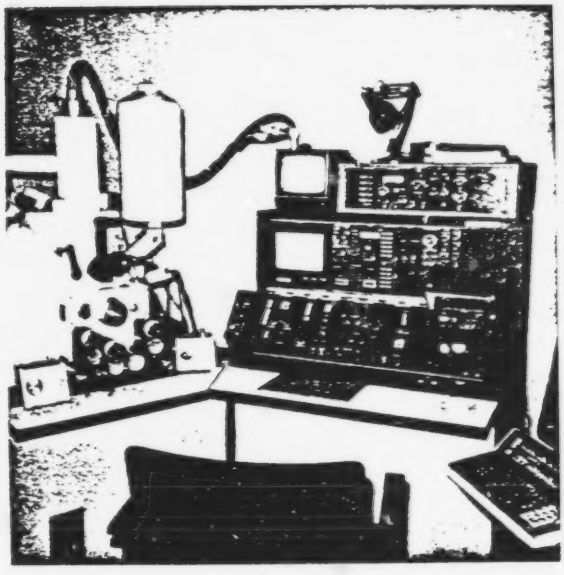


Fig. 6. Jeol 733 with Tracor Northern EDXA programmed to perform image analysis.

Figs. 7-12. Photomicrographs of Pb concentrate taken with Jeol 733.

Fig. 7. Topography

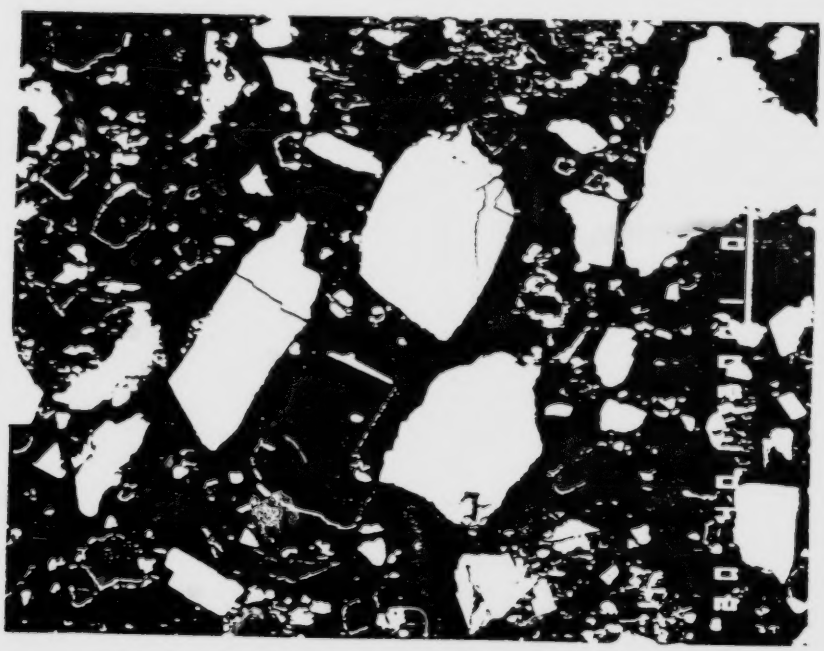


Fig. 8. BSE

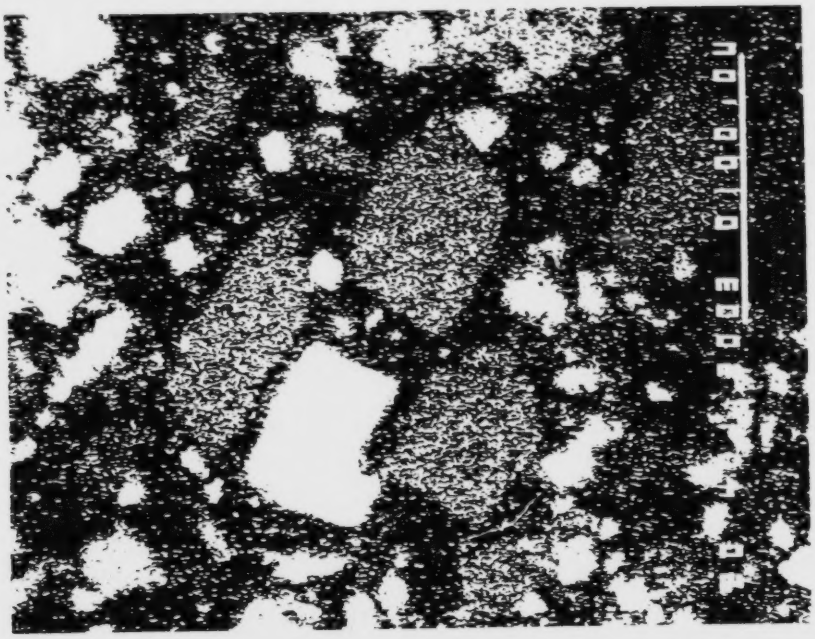


Fig. 9. FeKα

Fig. 10. ZnKα

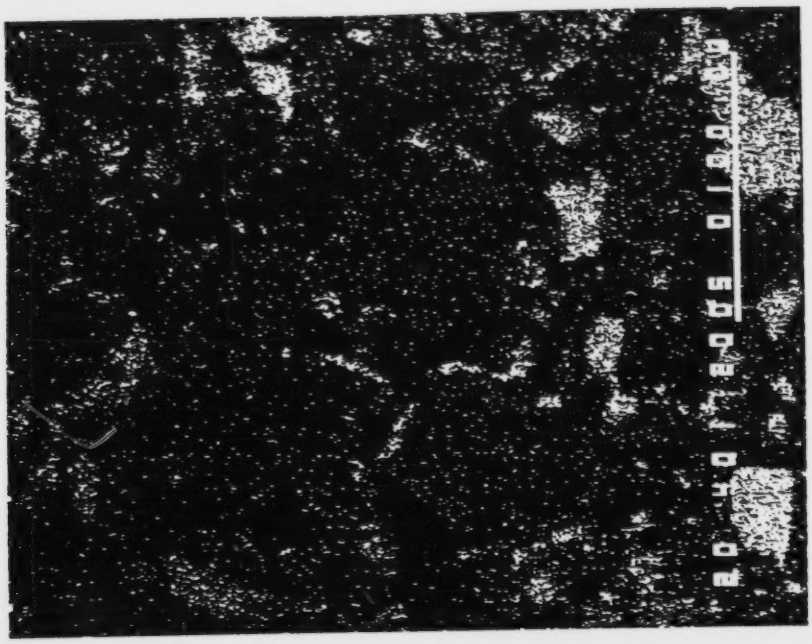


Fig. 11. PbKa

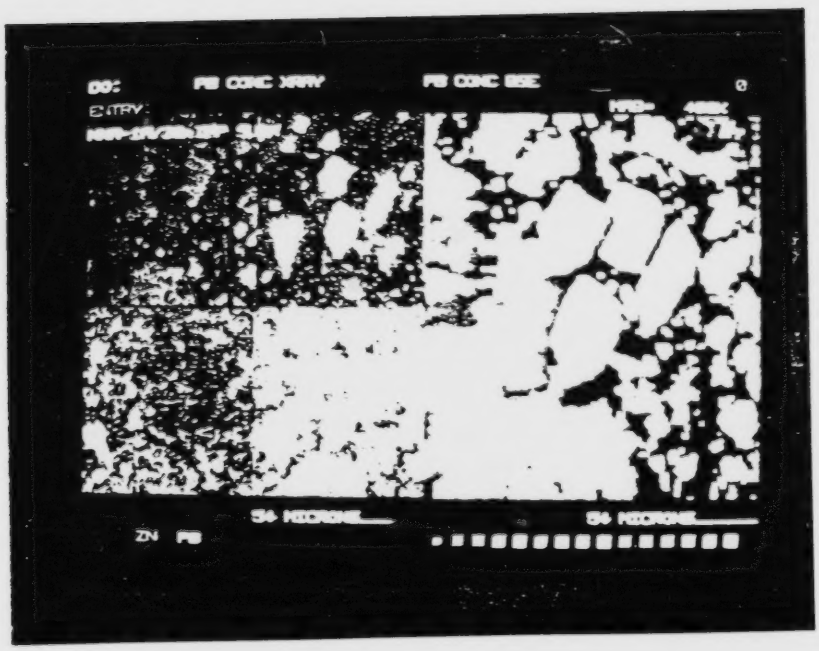


Fig. 12. FeKa, ZnKa, PbKa composite X-ray maps and BSE map

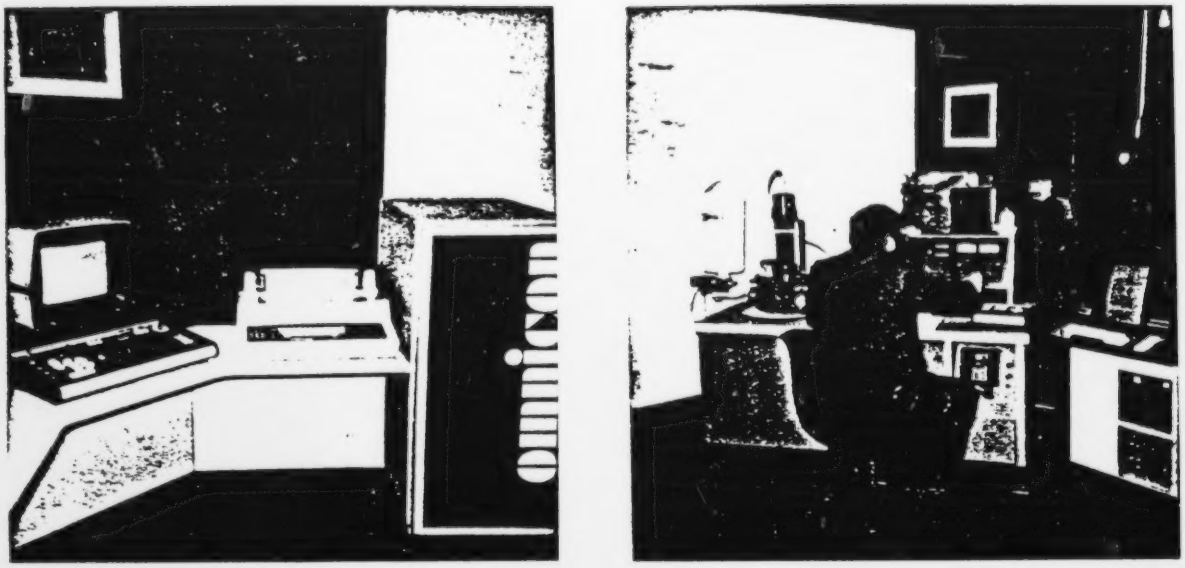


Fig. 13. Image analysis system at Bausch and Lomb. Right photograph: SEM and EDXA system with CRT control for EDXA (extreme right). Left photograph: Omnicon; cabinet with hardware and computer facilities, CRT and teletype.

APPENDIX A

Introduction

An SEM-EDXA based image analysis system that will be able to perform certain image analysis routines and to search for minerals in a polished or thin section is required. The system must be able to automatically analyse the particles in about 50 fields of 200 particles per field (about 10,000 particles per analysis) in about 3 to 5 hours. The required period of analysis is, therefore, 2 seconds or less per particle. The most sensitive B.S.E. detector available will be required so that mineral pairs that have small difference in atomic number can be discriminated. The detector will need at least two threshold pots so that three grey levels can be detected. The system will be programmed to automatically change from one grey level to the next or to combine two grey levels, in order to be able to do image analysis of free and unliberated grains.

I. General Particle Recognition and Characterization Program

Specific capabilities are desired from the particle recognition and characterization program. They are already present in one form or another on all the image analysis programs looked at to date, but minor modifications are required to meet our needs. They are:

1) freedom to easily set-up a chemistry file and to modify it;
2) freedom to alter the X-ray analysis statistics so that the operator can set-up for minimum length of time for X-ray analysis on each particle;
3) no lower limit for time of analysis for each particle since it is possible that minimum statistics for our need could be obtained in 0.1 seconds or less. We require the freedom to test and alter this to suit our sample and type of analysis;

4) ability to set and store the above three conditions and recall them for use as desired;

5) ability to do image analysis without X-ray analysis;

6) the image analysis output should be a hard copy printout with options to print out only (the wanted specified) data. The following is required:

   a) Summary Table -

      List of chemistry classes used in analysis. For each chemistry class give (1) number of particles, (2) percentage of surface area covered by the particles, (3) mean diameter and (4) wt%. (This will require a density input by operator for each chemistry class).

   b) Size distribution tables

      The size distribution tables should be printed out in two formats, each in a separate table. One table would give, for each chemistry class and for total particles, the percentage of surface area that is covered by particles within each size range. The other table would give number of particles within each size range.

      The size ranges are to be set by the operator rather than in a pre-set program, and the number of size ranges should be variable allowing up to about 12. It should be possible to store, recall and modify the size ranges set by the operator.

7) When required, it should be possible to obtain a printout for every particle in one or more chemistry classes. The printout should give, for each particle; area, mean diameter, maximum diameter, minimum diameter, aspect ratio, perimeter, shape factor ($\frac{P^2}{A}$) and location.

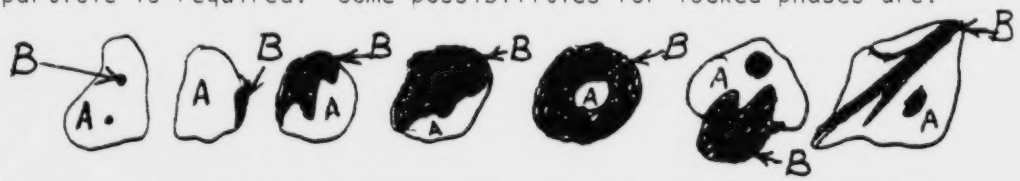
8) When required, it should be possible to obtain a printout for a miscellaneous chemistry class. The printout should give for each particle, the chemistry as well as area, diameter, and location.

9) Ability to return to a specific particle.

10) Ability to scan field and to get total area covered by detected particles without measuring each particle. Particles would be detected by backscatter, X-ray, or secondary.

II. Mineral Liberation Program - (a must for our application)

A program that will be able to detect and analyse locked phases in a particle is required. Some possibilities for locked phases are:



one phase (Phase B) would have one grey level on the backscatter, secondary electron or combined X-ray image and the other phase or phases (Phase A) would have another grey level. When this condition is met the identity of Phase B would have to be determined. The identity of Phase A is desirable, but frequently not necessary. Therefore, obtaining the identity of Phase A must be optional. The areas, diameters, and perimeter of Phase B and of the entire particle need to be measured. An option that might speed up analysis would be the freedom to request that the chemistry of Phase B, be determined as a first step. If Phase B has the specified identity, proceed with further analysis of the particle, if Phase B does not have the proper identity, move to next particle.

A sample of the printout data required for the Phase B individuals that have the required chemistry is:

Table A: area % - size distribution of host particle

size range µm (host particle)	area % of phase in particle								
	<5	5-10	10-25	25-50	50-75	75-90	90-95	>95	Total
<4.7	0								
4.7 - 6.5	0								
6.5 - 9.25	0.1								
9.25 - 13	0.12								
13 - 18.5	0.18								
18.5 - 26	0.1								
>26	0								
Total	0.5	1.5	5	8	12	14	8	51	100

Table B: Number of particles - size distribution of host particle

size range µm (host particle)	area % of phase in particle								
	<5	5-10	10-25	25-50	50-75	75-90	90-95	>95	Total
<4.7	0								
4.7 - 6.5	0								
6.5 - 9.25	30								
9.25 - 13	30								
13 - 18.5	10								
18.5 - 26	10								
>26									
Total	80	70	50	50	50	40	30	100	470

Table C: area % - size distribution of phase

size range µm (phase)	area % of phase in particle								
	< 5	5-10	10-25	25-50	50-75	75-90	90-95	>95	Total
<4.7	0.5								
4.7 - 6.5	0								
6.5 - 9.25	0								
9.25 - 13	0								
13 - 18.5	0								
18.5 - 26	0								
>26									
Total	0.5	1.5	5	8	12	14	8	51	100

Table D. number of individuals of phase - size distribution of phase

size range µm (phase)	area % of phase in particle								
	<5	5-10	10-25	25-50	50-75	75-90	90-95	>95	Total
<4.7	100								
4.7 - 6.5	0								
6.5 - 9.25	0								
9.25 - 13	0								
13 - 18.5	0								
18.5 - 26	0								
>26	0								
Total	100	90	85	70	65	50	35	100	595

Note: The size range would have more categories than shown in the tables.

Tables A and C show that 0.5% of the total surface area of Phase B is in particles that contain less tha 5% (surface) of the particle: 1.5% of the surface area of Phase B is in particles that contain 5-10% (surface area) of the particle. Table B gives numbers of host particles that fall within the size ranges and category of particle, and Table D gives numbers of Phase B individuals that are within the size ranges and categories of particles.

The size distribution data in Tables A and B gives the sizes in area % and number of particles respectively for the host particles containing Phase B. Tables C and D give the sizes in area % and in numbers of individuals respectively for Phase B in each category of particle.

III. Search Technique Program

Ability to scan a field and search for particles that are within a certain grey level and have a certain chemistry (one element or elemental pair). If the particle looked for has the required element, it should be

analysed to determine the chemistry, areas, diameters, and record location (field No., and location in field). Several hundred fields would have to be analysed. This technique would be used for three purposes where the concentration of a mineral is low.

1) To find particles of specified chemistry in a polished section that is about 1¼ inches in diameter. The particles would be 1 to 100 µm in diameter and the section would contain 1 to 20 particles of interest. A hard-copy printout would be required for each particle.
2) To determine % on basis of surface area, of particles having a specific chemistry. Obtaining this data would involve (i) measuring the total surface area of all particles in the field without analysisng the particles by X-ray. This would likely be done by scanning the field; (ii) analysis of particle with proper chemistry by X-ray and by image analysis. The printout would give area % of phase and, if required, liberation properties including size distribution.
3) Perform image analysis of particles that are present in small quantities. The printout would require liberation properties including size distributions.